

# En Chem, Inc.

## Quality Assurance Document

SET No: 1

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### STANDARD OPERATING PROCEDURE

TITLE: Florisil Cleanup of Samples for PCBs


DEPARTMENT: Semivolatile Organic Extractions

REFERENCES: Test Methods for Evaluating Solid Wastes SW-846  
Method 3620B, modified for PCBs.

#### PROCEDURE SUMMARY:

This procedure removes the majority of environmental and biological contaminants from PCBs in sample extracts. This operating procedure is applicable to any sample extract in hexane that requires additional cleanup before PCB determination. The sample extract is placed on a standardized Florisil column and eluted with petroleum ether.

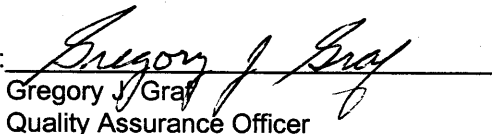
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### REAGENTS:

- . Petroleum ether, pesticide grade
- . Ethyl ether, without preservative, pesticide grade (optional)
- . Sodium sulfate, granular, anhydrous
- . Florisil, Fisher 60-100 mesh or equivalent
- . Aroclor 1016/1260

### APPARATUS:

- . Florisil columns, 19 mm id x 300 mm length with a 300-mL reservoir
- . 500-mL Erlenmeyer flasks or Kuderna-Danish concentrating apparatus
- . Assorted standard laboratory glassware

### PROCEDURE:

#### Preparation of Florisil

The Florisil may be used directly out of the container if validated.

#### Florisil Standardization

Each lot number of Florisil needs to be standardized. Once a lot has been standardized, it may be used following the same procedure that was used to standardize it, even if it is in a separate jar or box. Always check the Florisil validation file when using a new container of Florisil to be sure that the lot has been standardized.

The standardization procedure below exemplifies the typical procedure. The amount of Florisil used and the solvent volumes and mixtures may be changed to accommodate different characteristics the particular lot of Florisil may exhibit. For example, the solvent used for a particular lot of Florisil may need to have ethyl ether added (e.g. 1%) in order to elute some of the PCBs. Aroclor 1016/1260 is used for the standardization because it covers the whole range of PCBs to be analyzed. If the recovery of Aroclor 1016/1260 is at least 90%, and there are no clean-up related interferences, the Florisil is considered acceptable for use for PCBs.

1. Place a plug of glass wool into the bottom of two Florisil columns, one for a blank and one for the standard.
2. Add 20 g of Florisil to each column.
3. Top each Florisil column with 2 cm of sodium sulfate.
4. Pre-rinse each of the columns with 100 mL petroleum ether, taking the solvent down to the top of the sodium sulfate (do not let the column go dry). Discard the eluent.

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5. Label each column and a 500-mL Erlenmeyer flask for each.
6. Place a 500-mL Erlenmeyer flask under each of the Florisil columns (a Kuderna-Danish concentrating apparatus may be substituted for the Erlenmeyer if the sample is to be concentrated and exchanged).
7. To the blank column, add 5.0 mL of hexane, open the stopcock, and allow it to go down to the top of the sodium sulfate.
8. Close the stopcock.
9. To the standard column, add 5.0 mL of Aroclor 1016/1260 (at a concentration between 0.5 and 5.0 µg/mL in hexane), open the stopcock, and allow it to go down to the top of the sodium sulfate.
10. Close the stopcock.
11. Add 250 mL of petroleum ether to each column.
12. Open the stopcock and allow the solvent to flow at about 5 mL/minute until the flow stops.
13. Concentrate the eluent and exchange the solvent to hexane.
14. Analyze the eluted blank and standard using a gas chromatograph (GC) with a electron capture detector (ECD) and compare it with the Aroclor 1016/1260 used to standardize the Florisil. The blank should have no contaminants that will interfere with the analysis of the Aroclor 1016/1260. The recovery of the Aroclor 1016/1260, based on a direct comparison of the areas of five peaks from the standard to the same five peaks in the standard eluent, should be at least 90%. Make sure the peaks present in the analytical standards are present in the cleaned extract at the same ratios.
15. Record the lot number of the Florisil, the exact procedure, and the recoveries in the Florisil log book. Label all chromatograms and reports used in the GC analysis and keep them in the Florisil validation file.
16. If the above procedure does not provide at least 90% recovery, the eluting solvent may be changed by adding ethyl ether (e.g. 1%) or increasing the amount of petroleum ether used. The specific lot of Florisil may not be used for samples until an acceptable standardization procedure is completed.

Florisil Cleanup of Samples for PCBs

1. The sample extract should be in hexane.
2. Place a plug of glass wool in the bottom of as many Florisil columns as will be used to clean up blanks, samples, and spikes.
3. Complete Steps 2 through 6 of the standardization procedure.
4. Add 5.0 mL of the sample extract (in hexane) to the top of the column. Smaller volumes may be used if adjustments need to be made based on sample amount available.
5. Open the stopcock, allow the sample to go down to the top of the sodium sulfate, and close the stopcock.
6. Add 250 mL of petroleum ether to the column, open the stopcock, and allow the solvent to elute at a rate of about 5 mL/minute.
7. When all of the solvent has gone through the column, concentrate the sample extracts, exchange them to hexane, and dilute them to a needed volume.
8. The samples are ready for further cleanup or analysis, depending on the remaining matrix interferences.